

## Grinding characteristics and powder properties of the grains pre-treated with subzero temperature

学位名	博士(海洋科学)
学位授与機関	東京海洋大学
学位授与年度	2017
学位授与番号	12614博甲第443号
URL	<a href="http://id.nii.ac.jp/1342/00001458/">http://id.nii.ac.jp/1342/00001458/</a>

博士学位論文内容要旨  
Abstract

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論文題目 Title	Grinding characteristics and powder properties of the grains pre-treated with subzero temperature (氷点下温度で前処理した穀物の粉碎特性と粉の性状に関する研究)		

Grinding is an operation widely used in food industry, the most general method for cereal grain powder processing. In general, grinding is part of a larger set of operations involved in the process of size reduction. The size reduction has an influence on food processing such as extraction and drying. The particle size of food affects the yield of product such as oil and protein in grains; it also varies the duration for heat treatment such as blanching and cooking. Generally, the size reduction mechanism is comprised of deforming the food piece until it breaks. The particle of small size could be generated through the progress of erosion and breaking of outside part. Grinding to break cereal grains into flour can cause damage to starch granules, disruption of starch crystalline lamellae, and degradation of starch molecules. In addition, some grinding energy turns into heat in the grinder that can make the grain rubbery and difficult to break. The efficiency of energy consumption also considered as an important part of the grinding process. The grinding characteristics are affected by grain properties such as size, hardness, fibre and moisture content. The unit operations such as drying, soaking and freezing have been used to enhance the grinding efficiency and powder quality. Among the operations, the cereal grain treated with subzero temperature as pre-treatment become more brittle and easily broken. Thus, the powder production of smaller particle size can be easier than dry grinding. It also has advantages such as protection of heat labile components and reduction of grinding time. Freeze grinding is well-known method which realizes above mentioned advantages but it is difficult to control the temperature during grinding because grinders should be equipped with cooling device that leads to increase of production cost. From the viewpoint of practical use, low temperature pre-treatment prior to grinding is far more convenient but no previous study presented the effect of low temperature pre-treatment on grinding grains.

The objectives of this study were 1) to investigate the effect of pre-treatment at subzero temperature on the grinding characteristics of cereal grain, 2) to determine the physical properties of particle and bulk powder, 3) to evaluate the powder quality as processing raw material. The pre-treatment attempted in this study were freezing in the stocker of -20°C, -80°C and soaking into liquid nitrogen.

In Chapter 1, the grinding characteristics of grains (soybean, black soybean, buckwheat) were investigated in terms of particle size and energy consumption. In addition, the microscopic image of particles were observed. The average particle size of ground soybean, black soybean and buckwheat powders decreased as pre-treatment temperature decreased. The constants derived from Bond's law that described grinding characteristics revealed that the freezing as pre-treatment is effective on grinding process. In all grain samples, the Bond's constant and work index showed lower values as the pre-treatment temperature decreased. The scanning electron microscopy was used for observation of surface damages on the particles by grinding process. Some cracks were seen on the surface of particles of soybean powder ground with freezing as pre-treatment. On the other hand, the particles of black soybean powder showed no fractures. The buckwheat powder showed the sharp breakage surfaces, especially particles made by dry grinding (without pre-treatment) had the damaged starch granules structure. The freezing as pre-treatment of grains prior to grinding process was found to be effective to control their grinding characteristics and microstructure damages. This chapter showed that decreasing the temperature of soybean, black soybeans and buckwheat was valuable to control their grinding characteristics, including

energy consumption and average particle size.

In Chapter 2, the bulk powder properties of buckwheat powder made from the seed pre-treated at subzero temperature was focused. Particle size and its distribution and resulted flowability of bulk powders were investigated. The mean particle size of powders from pre-treated buckwheat seed was smaller than that of control (without pre-treatment). As the grinding time increased, the particle size decreased exponentially. At the condition of grinding time equals to 120 s, particle size distributions concentrated to specific particle size (smaller size). Bulk of buckwheat powder showed a general flow pattern and the flow became cohesive with increasing grinding time. Buckwheat powder made from pre-treated seed was found to approach closer to the cohesive flow. It was found the pre-treatment on the buckwheat powder was effective to size reduction significantly. The flow characteristics evaluated in this chapter provided important information to predict the flow pattern for buckwheat powders.

In Chapter 3, the buckwheat powder qualities were evaluated. The microscopic image of control particles showed damaged starch granules structure. On the other hand, the particle made by grinding with pre-treatment showed smooth surface of particles with less broken part. The particles made from pre-treated buckwheat had more sharp breakage surface than the control particles. In addition, the degree of damaged starch on the particle made from pre-treated sample was less than control (without pre-treatment). The flavor compounds abounded more in buckwheat particles ground with freezing as pre-treatment. Just after the grinding, the flavor components of buckwheat powders were almost independent of grinding conditions. After the 6 months storage, however, buckwheat powder with pre-treated at subzero temperature showed more flavor compounds retained than control. This chapter demonstrated that the grinding process of buckwheat with pre-treated at low temperature is useful for reducing their microstructural damage and retaining flavor component after the storage.

In Chapter 4, the quality of buckwheat powder as processing raw material was investigated. Differential scanning calorimeter (DSC) was used for determination of gelatinization characteristics of buckwheat powder. Control powder (without pre-treatment) shows lower values of thermal properties due to high level of damaged starch content. The viscoelastic behavior of buckwheat dough was investigated by dynamic rheometer. The storage modulus ( $G'$ , elastic property) and the loss modulus ( $G''$ , viscous property) of the dough made of the buckwheat powder ground with freezing pre-treatment showed lower levels than control (made of powder ground without pre-treatment). However, the texture property of boiled buckwheat dough was not changed by pre-treatment. It was confirmed freezing pre-treatment resulted in a lower content of damaged starch that could improve the quality of final product.

In conclusion, this study demonstrated the effect of pre-treatment with subzero temperature on the grain grinding process and powder properties. The quality of powder was evaluated and the bulk powder properties were determined. The pre-treatment at subzero temperature significantly affected the physical property of grains. It also was effective in terms of energy consumption during grinding. The grain pre-treated with subzero temperature enabled less damage during grinding. The particle size, shape, and bulk powder behavior (flowability) were affected by pre-treatment of grain. In addition, the final product qualities made of the powder were expected to be improved. Consequently, this study provided important information to apply the pre-treatment at subzero temperature for grain powder processing.